Propulsion and Power Research 2013;2(3):194–200

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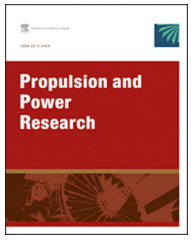


ORIGINAL ARTICLE

# Role of electrodes in ambient electrolytic decomposition of hydroxylammonium nitrate (HAN) solutions


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Received 13 April 2013; accepted 17 June 2013
Available online 27 August 2013

**KEYWORDS**

Electrolytic decomposition;
Hydroxylammonium nitrate (HAN);
Copper wire;
Inert carbon rod;
Electrode

**Abstract** Decomposition of hydroxylammonium nitrate (HAN) solution with electrolytic decomposition method has attracted much attention in recent years due to its efficiencies and practicability. However, the phenomenon has not been well-studied till now. By utilizing mathematical model currently available, the effect of water content and power used for decomposition was studied. Experiment data shows that sacrificial material such as copper or aluminum outperforms inert electrodes in the decomposition of HAN solution. In the case of using copper wire to electrolyse HAN solutions, approximately 10 seconds is required to reach 100 °C regardless of concentration of HAN. In term of power consumption, 100 W–300 W was found to be the range in which decomposition could be triggered effectively using copper wire as electrodes.



## 1. Introduction

Hydroxylammonium nitrate (HAN), with the chemical formula $NH_3OHNO_3$, has been widely recognized as a liquid gun material and rocket propellant due to its unique advantages such as high energy density, low handling and low storage cost. The relatively high specific impulse and clean post-combustion gas species has made HAN known to be a great oxidizer for hybrid rockets [1–3]. However, several crucial challenges, such as combustion stability

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Peer review under responsibility of National Laboratory for Aeronautics and Astronautics, China.

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http://dx.doi.org/10.1016/j.jppr.2013.07.002

## Nomenclature

| | |
|---|---|
| A | ampere |
| A.E | activation energy |
| Al | aluminium |
| aq | aqueous |
| Cu | copper |
| $Cu(NO_3)_2$ | copper nitrate |
| $Cu^{2+}$ | copper (II) ion |
| *d* | diameter of copper wire |
| DC | direct current |
| $e^-$ | electron |
| $H^+$ | hydrogen ion |
| $H_2$ | hydrogen |
| HAN | hydroxylammonium nitrate |
| $HNO_3$ | nitric acid |
| HONO | nitrous acid |
| l | liquid |
| $(NH_3NO_3)^+NO_3^-$ | HAN |
| $N_2O$ | nitrous oxide |
| NO | nitrogen oxide |
| $NO_2$ | nitrogen dioxide |
| NOx | mono-nitrogen oxides |
| $O_2$ | oxygen |
| PDMS | polydimethyl siloxane |

and reliable ignition or combustion method to provide high energy output need to be resolved, in order to further develop other potential usage of HAN in various applications ranging from space transport to daily micro-energy resources for microreactors [4].

Extensive studies relevant to HAN have been conducted by many researchers including the combustive behavior [5,6] and thermal heating or catalytic ignition [7–9]. The process resulted in huge energy loss from ignition, which is particularly more significant at the microscale due to the large surface-to-volume ratio of combustion chamber.

Compared to other common energetic materials, decomposition of HAN-based material is more complicated due to the evolutions of multi-gas species involved in various stages throughout the process [10]. Different models of chemical reactions in decomposition of HAN-based materials were developed using different techniques. A study by Klein et. al. concluded that the combustion of a HAN-triethanol ammonium nitrate (TEAN) solution is completed with three stages in sequence of initiation, ignition, followed by combustion [11]. This finding is supported by Oxley and Brower in which a similar trend of three stages combustion mechanism were proposed [12]. However, the formation of NOx gas species from the thermal combustion process of HAN-based solution by Oxley and Brower remains as a centre of argument as other species such as HONO and $N_2$ are favored. Meanwhile, Lee and Litzinger summarized the above reaction mechanisms and constructed a theoretical reduced reaction model in order to deduce the Arrhenius-type reaction rates for binary system of HAN decomposition [13]. The reduced model improved the overall chemical reactions involved by introducing intermediate reactions such as formation of HONO and NOx.

Having considered the limitations of the conventional combustions techniques, a relatively new ignition technique with electrolysis was developed, in which a direct current (DC) is directly deposited into the solution to trigger ignition. The overall combustion process is proven to be more efficient with reduced heat loss [14,15]. Yetter et al. concluded that any reaction mechanism that competes with proton transfer reaction, which initiates the following exothermic reactions, should decrease the reaction temperature, thus increase the reaction rates [16]. In addition to the advantage to a lower onset temperature in electrolytic decomposition of HAN solution, the additional formation of hydrogen and oxygen gases from electrolysis process were believed to enhance the ignition and combustion [17].

In this paper, investigations into the use of sacrificial electrodes to replace conventional inert electrodes were carried out. In addition, the electrolytic decomposition of HAN solution was also studied using low DC power in which the parameters include the effects of power supply to various concentrations of HAN solutions.

## 2. Theory of HAN decomposition

Thermal and electrolytic decomposition paths of HAN solution were compared in Table 1. In thermal decomposition, water evaporation, which is the chemical rate determining step, would take place, before ignition caused by reaction between nitric acid and hydroxylamine. In the decay stage, six simplified kinetic equations occurred simultaneously were proposed. The decomposition via electrolysis of ignition and decay stages are similar to that of HAN thermal decomposition process. However, the only main difference that makes electrolytic decomposition an improved technique was the initiation process. Electrolytic decomposition utilized the charge-rich characteristic of HAN solution to initiate decomposition by stimulating electrons transfer within HAN solution via electrolysis to reach its excited stage, before further reactions take place as described in previous section.

## 3. Experiment

### 3.1. Preparation of HAN solutions

HAN-water mixture was prepared in house via titration between aqueous solution of hydroxylamine (50%wt) and nitric acid (35%wt, both from Sigma-Aldrich, US). The solution was continuously kept in an ice water bath throughout the experiment to avoid self-decomposition, until pH = 2.50. The solution was then further purified in a rotary evaporator (Laborota 4003, Heidolph, Germany) to achieve HAN solutions with 20%-80% of mass fraction.

**Table 1** Comparison of reaction mechanism using different HAN decomposition techniques.

| Thermal decomposition (Lee and Litzinger) [13] | | Electrolytic decomposition (Yetter and Young) [16,17] |
|---|---|---|
| A.E/(kJ/mol) | Induction: | Initiation: |
| 65.314 | 1. $HAN + H_2O \rightarrow NH_2OH + HNO_3 + H_2O$ | At anode,<br>$H_2O \rightarrow 0.5O_2 + 2\,H^+ + 2e^-$<br>$(NH_3OH)^+NO_3^- + H^+ \rightarrow NH_3OH^+ + HNO_3$<br>At cathode,<br>$2NH_3OH^+ + 2e^- \rightarrow 2NH_2OH + H_2$<br>Hence, overall reaction is<br>$H_2O + 2(NH_3OH)^+NO_3^- + H^+ \rightarrow 2NH_2OH + H_2 + 0.5O_2$ |
| | Ignition: | |
| 30.564 | 2. $NH_2OH + HNO_3 \rightarrow HONO + HNO + H_2O$ | |
| | Decay: | |
| 14.654 | 3. $NH_2OH + HONO \rightarrow N_2O + 2\,H_2O$ | |
| 11.723 | 4. $NH_2OH + HNO \rightarrow N_2 + 2\,H_2O$ | |
| 38.937 | 5. $3HONO \rightarrow 2NO + HNO_3 + H_2O$ | |
| 72.013 | 6. $2HNO \rightarrow N_2O + H_2O$ | |
| 69.920 | 7. $HNO + HNO_3 \rightarrow 2HONO$ | |
| 3.349 | 8. $HONO + HNO_3 \rightarrow 2NO_2 + H_2O$ | |

### 3.2. Electrolytic decomposition

5 ml of HAN solution were poured into a 50 ml beaker, followed by the insertion of the commercially available copper wire ($d = 2.8$ mm, BS 6231, Element14, Malaysia) that served as electrodes. The beaker was insulated and covered with a custom-made polymer, PDMS lid with four 0.5 mm diameter holes, punched for sensors insertion. K-type thermocouple was immersed into the solution to record temperature changes along decomposition process while the gas analyzer (TESTO 340, USA) and thermohygrometer (37953-20, Cole-Parmer, US) were used to record the gas species released as well as change in moisture content respectively. DC power supply from a DC power unit (Elektro-Automatik, Germany) was switched on for 2 minutes or until the solution in the beaker used up. The copper wires were replaced after each experiment and the distance between copper wires in the beaker was always kept between 3 cm-4 cm.

The experiment was repeated using aluminum foil (Superking Aluminium, China) and carbon rod electrodes (MiniScience, USA). All experiments were repeated at least three times for reliability and accuracy purpose. Average results were compared with theoretical kinetics as discussed in previous section. Since the beaker is an enclosed system, the increment of moisture content and growth of NOx gases were assumed to be solely due to the HAN decomposition. The schematic experimental set up is shown in Figure 1.

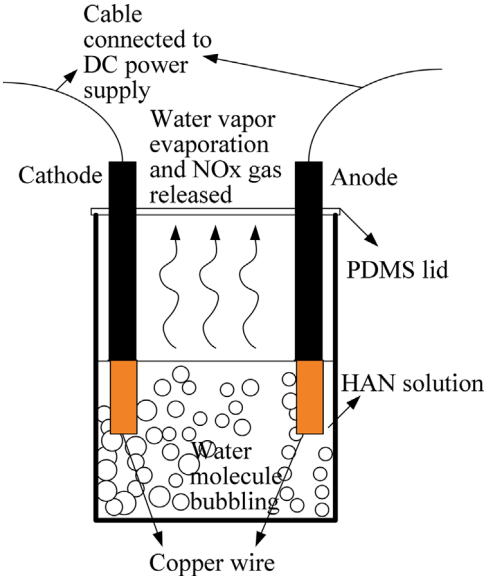


**Figure 1** Illustration of HAN electrolytic decomposition process.

## 4. Results and discussions

### 4.1. Electrolytic decomposition of HAN solution using copper wire

In this experiment, 70%wt HAN solution was chosen as a benchmarking for electrolytic decomposition as it is the threshold concentration for complete decomposition with no condensed phase formed at post combustion [18].

Immediately after power supply into the HAN solution was switched on, mild reaction took place as the solution turned from transparent into blue color solution with immediate sizzling sound and continuous bubbling. This indicates the stage of initiation and ignition of decomposition. Detection on the presence of NOx, as shown in Figure 2, indicates that the decomposition was progressing into the decay stage. The initial sharp increment of NO species can be attributed to the oxidization of $N_2O$ intermediate species that were further oxidized into $NO_2$, a more stable species compared to $N_2O$. Decomposition of $NO_2$ back into NO and $O_2$ at later stage was not favored due

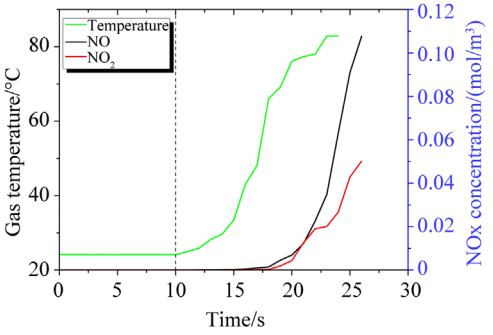


**Figure 2** Temperature profile of gases and variation in concentrations of NOx for decomposition of 70%wt HAN decomposition at 300 W (60 V, 5 A). Section 1 refers to the initiation and ignition while Section 2 is the decay stage in which post combustion gas species were released. Since electrolysis is known to be an endotermic process and no external heart provided, drastic rise in the solution temperature for 15 seconds was contributed by the energy released from the decomposition of HAN molecules.

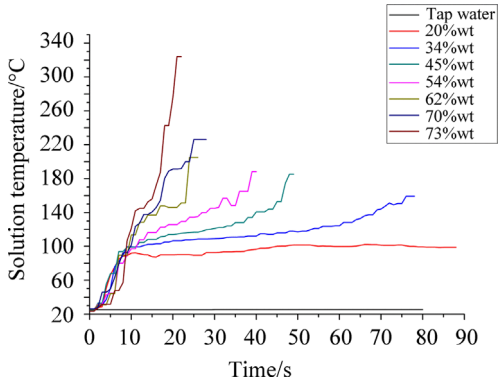


**Figure 3** HAN solution temperature profile of electrolytic decomposition using copper wire as electrodes at various concentration levels of HAN at 300 W (60 V, 5 A). Electrolysis of tap water shows temperature rise in the solution caused solely by the decomposition of HAN molecules.

to insufficiency of energy since the process will only occur at a measureable rate higher than 200 °C. In addition, NO was also generated through the chemical reaction between the copper anode with nitric acid.

$$3Cu(s) + 8HNO_3(aq) \rightarrow 3Cu(NO_3)_2(aq) + 2NO(g) + 4H_2O(l) \quad (1)$$

### 4.2. *Effect of electrode selections*

The use of copper electrode created a competitive environment during initiation stage because both copper and water undergo oxidation at anode with oxidation of copper electrode favored ahead of water due to its relatively lower standard electrode potential. When power supply was switched on, electrolytic decomposition of water and HAN takes place simultaneously. The previous case is an endothermic reaction while the latter is an exothermic reaction, which contributed to the rise in temperature of the solution. As shown in Figure 3, the process can be separated into two main sections, i.e. initiation and decomposition.

The experimental result shows that initiation of HAN electrolytic decomposition is independent of its concentration as approximately 10 seconds were required for all solutions to reach 100 °C, the boiling point of water at standard condition. The solution near to the anode started to turn into light blue which indicates the occurrence of $Cu^{2+}$ ion. Excess electrons contributed by copper electrode accelerated the reaction in cathode as the electrons supply increased by two fold, which hasten the overall chemical reaction. The governing chemical reactions are shown as below:

At anode,

$$H_2O(1) \rightarrow 0.5O_2 + 2H^+ + 2e^- \quad (2)$$

$$Cu \rightarrow Cu^{2+} + 2e^- \quad (3)$$

$$(NH_2OH)^+NO_3^- + H^+ \rightarrow NH_3OH^+ + HNO_3 \quad (4)$$

At cathode,

$$2(NH_3OH)^+ + 2e^-_{water} + 2e^-_{copper} \rightarrow 2NH_2OH + H_2 \quad (5)$$

Hence, the overall reaction is

$$H_2O + 2(NH_3OH)^+NO_3^- \rightarrow 2HNO_3 + 2NH_2OH + H_2 + 0.5O_2 \quad (6)$$

However, the decomposition phenomenon behaves distinctive after initiation step. This can be observed by mild increment of temperature profile for diluted HAN solution (20%wt-34%wt), due to low HAN content in the solution that energy released was only used for heating the solution. Hence, both solutions can hardly undergo complete decomposition within 2 minutes of experiment with liquid residue left in beaker. As the mass fraction of HAN increases, more energy released by the decomposition of HAN was absorbed by water molecules, results in the water loss through electrolytic decomposition as well as boiling simultaneously. At moderate concentrated HAN solution (45%wt-62%wt), heat released surpassing heat absorbed by water molecules, causing higher rate of escape of water molecules from liquid into gas phase, followed by a sudden temperature surge just before the solution sample used up. Temperature of the solution increases significantly when water content in the solution decreases. At higher concentration (73%wt of HAN), large amount of energy released with only little water to absorb it to cause the temperature of the solution increased in a short interval. Besides, concentrated HAN solution underwent a relatively rapid decomposition process (≈28 seconds) which is extremely crucial in application with limited on board volume for fuel storage and quick combustion performance response such as

microsatellite fuel system control in which high energy density fuel is always favored.

Since aluminium is a common element in solid propellant currently in use, copper electrode was replaced with aluminium foil to investigate its efficiency as electrodes.

Decomposition phenomena of HAN solution using aluminium foil as electrodes are slightly different from the previous case. As shown in Figure 4, the HAN solution temperature would only rise above 100 °C when the mass fraction of HAN exceeding 34%wt with 20 seconds required. For concentrated HAN solution, the temperature rise profile was identical to the previous case of using commercial copper wire to serve as electrode–approximately 10 seconds to achieve solution temperature of 100 °C. Aluminium offers great electrons transfer during initiation stage, in which the oxidation of aluminum can occur without additional external energy supply, due to the fact that both copper and aluminium undergo similar electron transfer mechanism. Copper is known as a better electric conductor as its electricity conductance performance is approximately 55% better than the aluminium in identical cross sectional area, due to higher electron-phonon coupling effect of aluminium [19]. At high temperature, electron-phonon coupling effect in aluminium is more significant, in which the electron transfer from the aluminium metal body to surrounding is reduced.

Initiation reaction mechanism based on aluminium electrode:

At anode,

$$H_2O(l) \rightarrow 0.5O_2 + 2H^+ + 2e^- \quad (7)$$

$$Al \rightarrow Al^{3+} + 3e^- \quad (8)$$

$$(NH_3OH)^+NO_3^- + H^+ \rightarrow NH_3OH^+ + HNO_3 \quad (9)$$

At cathode,

$$2(NH_3OH)^+ + 2e^-_{water} + 3e^-_{Al} \rightarrow 2NH_2OH + H_2 \quad (10)$$

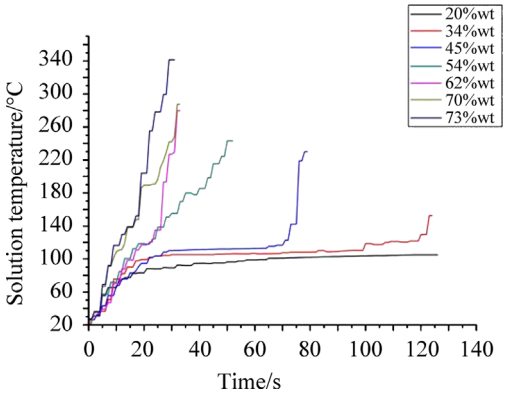


**Figure 4** HAN solution temperature profile of electrolytic decomposition using aluminum foils as electrodes at various concentration levels of HAN at 300 W (60 V, 5 A).

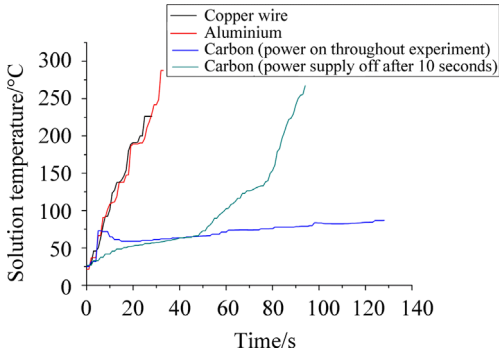


**Figure 5** Comparison on effect of electrodes between copper wire and carbon rod at electrolytic decomposition of 70%wt HAN at 300 W (60 V, 5 A). While the use of aluminium and copper electrodes were almost identical. The effect of carbon electrodes is notably and predictable on autocatalytic reaction of HAN solution.

Hence, the overall reaction is

$$H_2O + 2(NH_3OH)^+NO_3^- \rightarrow 2HNO_3 + 2NH_2OH + H_2 + 0.5O_2 \quad (11)$$

Result from Figure 5 shows complete deviation of decomposition behavior when inert carbon electrodes were used as electrodes. Temperature of HAN solution remained almost constant throughout the experiment with no phenomenon of decomposition observed when DC power was supplied continuously for 2 minutes due to aromaticity of carbon electrode, in which electrons can move freely within the plane of carbon electrode layers instead of across the eletrodes, which halted the completion of circuit and caused decomposition to fail.

In a different experiment in which power supply was cut off after 10 seconds of operation, it was astonishingly found that HAN solution would undergo autocatalytic decomposition for a relative long interval.

The major difference in decomposition behavior based on solution temperature profile using copper wire, aluminum foil and carbon electrodes can be attributed to different electron transfer mechanism during eletrolysis process. Compared to carbon electrode, copper wire and aluminium foils that served as sacrificial electrodes are more reactive, constantly providing eletrons into the solution by oxidizing the metals into ions throughout the process. This is further supported by change in the color of the residue solution, formation of crystal-like solid and mass change in both cathode and anode after each experiment. Although effective, the phenomenon hinders the long term application of sacrificial electrodes as multiple decomposition is not achievable with rapid mass decay in the anode. Furthermore, the formation of crystal-like solid would potentially create other issues such as decomposition chamber blockage and wall surface damaged. A tradeoff between decomposition delay and the abovementioned issues also needs to be taken into consideration prior to electrode selection for electrolytic decomposition process.

As observed in Figure 6, there is no definite trend on temperature profile in decomposition of HAN solution by using inert carbon rod as electrode. HAN at 45%wt is the threshold concentration in order for autocatalytic decomposition process to take place in a short interval.

### 4.3. *Effect of power input*

Second column in Table 1 indicates importance of rate of electron transfer in electrolytic decomposition of HAN solution, in which the intermediate chemicals released in cathode for further decomposition into gas species. As shown in Figure 7, the threshold power needed to effectively trigger electrolytic decomposition is 90 W (60 V, 1.5 A). Low input power such as 30 W (60 V, 0.5 A) and 60 W (60 V, 1 A) is insufficient for decomposition of HAN solution due to competition between HAN and water molecules. Substantial growth of temperature slope was observed when power input was increased from 60 W to 180 W. As the power input increased continuously, the influence towards decomposition process was limited by the presence of ions available in HAN solution.

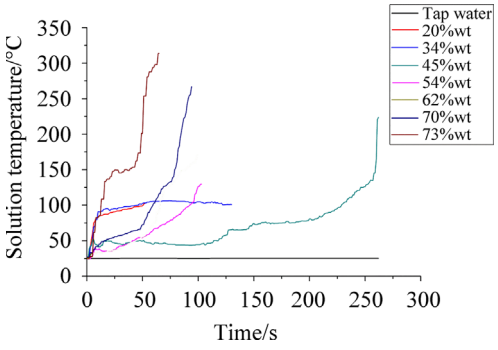


**Figure 6** HAN solution temperature profile of autocatalytic decomposition using carbon electrode at various concentration levels of HAN at 300 W (60 V, 5 A). HAN at low concentration (20%wt-34%wt) requires long time to undergo autocatalytic decomposition due to high content of water, which consumes most of the output energy through water evaporation.

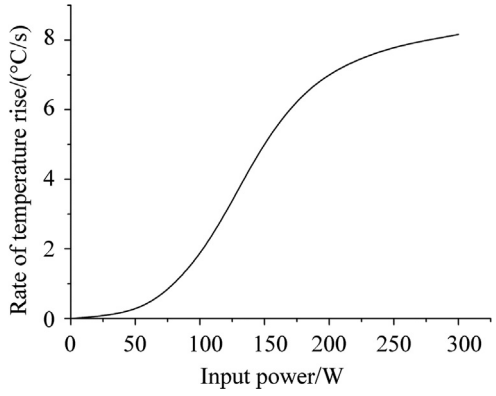


**Figure 7** Solution temperature profile of HAN decomposition process using copper wire at different power input applied across 70%wt HAN solution.

Despite the ease-execution of electrolytic decomposition technique, various disputes remain unclear especially the role of electrodes in electrolytic decomposition process. The effect of power input and concentration of HAN towards decomposition phenomenon also has no genuine studies on these areas to our best understanding. Copper wire has different electrode potential behavior in low (micromolar) and high (supermicromolar) acidic solution concentration in which Nernst equation is the governing equation for high concentration electrolysis process. This is different from lower concentration (sub-micromolar) since the adsorption equilibrium is predominant, and the electrode potential is related to the bulk concentration through adsorption equilibrium, which determines the surface concentration and the electrode potential. However, the above finding does not provide solution for some uncertainties such as the effect of continuous rises of solution temperature towards electrolysis process and the solubility as well as its behavior of copper ion in acidic environment. In addition, other vital factors remain unresolved such as condensation and solubility of NOx into HAN solution at high temperature.

In addition, this energy-saving decomposition mechanism which requires an in-depth understanding of combined electrolytic and thermal reaction mechanism is still absent in literature. Parameters that can reduce reaction delay such as residence time between reactions and conversion rate of the chemical equations that can potentially further harvest extra energy from the chemical reactions is not fully understood, hence giving uncertainties in analyzing the overall decomposition process.

## 5. Conclusions and future works

In this paper, we concluded selection of electrodes is an important role as the performance of conductivity is a determining factor. Although carbon was commonly used as electrodes in most electrolysis, it is not an ideal candidate in the decomposition of HAN solutions. Instead, sacrificial materials such as copper and aluminum are more efficient for the purpose. Copper wire electrodes would shorten initiation process at a constant interval for decomposition delay when 300 W is used. Although water a determining factor for its performance, all concentration of HAN solutions take about 10 seconds to heat up to 100 °C, which is the boiling point of water at standard condition. By electrolyzing 73%wt HAN solution with aluminum foil, solution temperature as high as 320 °C was recorded. In addition, the decomposition delay was found to increase up to ≈200 seconds when inert carbon rod electrodes were used to electrolyse low concentration HAN solutions. Experiment also shows that in term of power consumption, 100 W–300 W was found to be the range in which decomposition could be triggered effectively. Further increase in power supply may have limited effect on the rate of decomposition.

The research work is currently extending into the search of a suitable electrode that can enhance decomposition process without itself being consumed along the process and establishing model or scaling law which can relate electrochemical properties of the electrodes to chemical properties of HAN solution.

## Acknowledgements

The authors would like to acknowledge Ministry of Science, Technology and Innovation Malaysia (MOSTI) eScience Fund for funding the research project (04-05-05-SF0008). In addition, the authors would also like to thank Professor Charles Kappenstein from the University de Poitiers, France for his insightful advice towards the experiments.

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